EAST Search History

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	12934	membrane and aniline or polyaniline	USPAT; EPO; DERWENT	AND	ON	2006/08/22 12:21
L2	308	1 and gold same particles	USPAT; EPO; DERWENT	AND	ON	2006/08/22 12:21
L3	42	2 and nanoparticles	USPAT; EPO; DERWENT	AND	ON	2006/08/22 12:22

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TITLE: Polymer-inorganic particle composites

DATE-ISSUED: April 19, 2005

INVENTOR-INFORMATION:

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APPL-NO: 10/422149

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PARENT-CASE:

CROSS REFERENCE TO RELATED APPLICATIONS

This application is a divisional of U.S. patent application Ser. No. 09/818,141, now U.S. Pat. No. 6,599,631 to Kambe et al., filed on Mar. 27, 2001, entitled "Polymer-Inorganic Particle Composites," incorporated herein by reference, which claims priority to U.S. provisional application Ser. No. 60/265,169 filed on Jan. 26, 2001, entitled "Polymer-Inorganic Particle Composites," incorporated herein by reference.

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TYPE  IPC          DATE
CIPS  C08K9/00 20060101
CIPS  C08K9/08 20060101
CIPS  C08G18/00 20060101
CIPS  B32B27/08 20060101
CIPS  C08G18/38 20060101
CIPS  C08G83/00 20060101
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See application file for complete search history

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ART-UNIT: 1712

PRIMARY-EXAMINER: Peng; Kuo-Liang

ATTY-AGENT-FIRM: Patterson, Thuente, Skaar & Christensen P.C.

ABSTRACT:

Inorganic particle/polymer composites are described that involve chemical bonding between the elements of the composite. In some embodiments, the composite composition includes a polymer having side groups chemically bonded to inorganic particles. Furthermore, the composite composition can include chemically bonded inorganic particles and ordered copolymers. Various electrical, optical and electro-optical devices can be formed from the composites.

25 Claims, 49 Drawing figures

Exemplary Claim Number: 1

Number of Drawing Sheets: 23

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Detailed Description Text - DETX (2):
   Composite or hybrid materials generally are formed by chemically bonding a linker compound with both an inorganic **nanoparticle** and a monomer/polymer unit, although in some embodiments, the polymer is directly bonded to.the inorganic particle. Through the use of the linker compound, stable, uniform polymer-inorganic particle composites can be formed with highly dispersed inorganic particles. Specifically, high particle loadings can be achieved without agglomeration of the particles, provided that the particles are functionalized with groups that do not easily bond to themselves, which can result in the formation of hard agglomerates. The composite may represent a synergistic effect of the combined component. The advantages of the combination can be structural, electronic or optical.

Detailed Description Text - DETX (9):
   The inorganic particles generally include metal or metalloid elements in their elemental form or in compounds. Specifically, the inorganic particles can include, for example, elemental metal or elemental metalloid, i.e. un-ionized elements, metal/metalloid oxides, metal/metalloid nitrides, metal/metalloid carbides, metal/metalloid sulfides or combinations thereof. Metalloids are elements that exhibit chemical properties intermediate between or inclusive of metals and nonmetals. Metalloid elements include silicon, boron, arsenic, antimony, and tellurium. Preferred particles have an average diameter of less than about 500 nanometers (nm). Suitable **nanoparticles** can be formed, for example, by flame synthesis, combustion, or sol gel approaches. Preferred methods for synthesizing the particles include laser pyrolysis in which light from an intense focused source drives the reaction to form the particles. Laser pyrolysis is useful in the formation of particles that are highly uniform in composition, crystallinity and size.

Detailed Description Text - DETX (21):
   To form high index-of-refraction materials, high particle loadings are generally used. The index-of-refraction of the composite is expected to be approximately a linear combination by the volume ratios of index-of-refraction of the inorganic particles and the polymer. The use of **nanoparticles** has the advantage for optical materials of higher transparency and reduced scattering of light relative to larger inorganic particles, especially effective in

reducing scattering in the infrared portion of the electromagnetic spectrum including wavelengths of about 0.8 microns to about 5.0 microns.

Detailed Description Text - DETX (40):

Suitable polymers include organic polymers, silicon based polymers and other inorganic polymers. Many different types of polymers are suitable as long as they have terminal groups and/or preferably side groups capable of bonding to a linker. Suitable organic polymers include, for example, polyamides (nylons), polyimides, polycarbonates, polyurethanes, polyacrylonitrile, polyacrylic acid, polyacrylates, polyacrylamides, polyvinyl alcohol, polyvinyl chloride, heterocyclic polymers, polyesters and modified polyolefins. Composites formed with nylon polymers, i.e., polyamides, and inorganic **nanoparticles** can be called Nanonylon.TM.. Suitable polymers include conjugated polymers within the polymer backbone, such as polyacetylene, and aromatic polymers within the polymer backbone, such as poly(p-phenylene), poly(phenylene vinylene), **polyaniline,** polythiophene, poly(phenylene sulfide), polypyrrole and copolymers and derivatives thereof. Some polymers can be bonded to linkers at functional side groups. The polymer can inherently include desired functional groups, can be chemically modified to introduce desired functional groups or copolymerized with monomer units to introduce a portions of desired functional groups. Electrically conducting polymers can be particularly useful for certain applications. Polyacetylene becomes an electrical conductor upon doping with electron acceptors, such as halogens, or electron donors, such as alkali metals. Mixtures of polymers can also be used, although in many embodiments one of the above polymers is present in at least about 50% by weight, optionally at least about 75% by weight and optionally at least about 90% by weight of the polymer/monomer composition. Similarly, some composites include only a single polymer/monomer composition bonded into the composite. Within a crosslinked structure, a polymer is identifiable by 3 or more repeat units along a chain, except for hydrocarbon chains which are not considered polymers unless they have a repeating side group or at least about 50 carbon-carbon bonds within the chain.

Detailed Description Text - DETX (50):

Generally, thiol groups can be used to bind to metal sulfide **particles** and certain metal **particles, such as gold,** silver, cadmium and zinc. Carboxyl groups can bind to other metal **particles,** such as aluminum, titanium, zirconium, lanthanum and actinium. Similarly, amines and hydroxide groups would be expected to bind with metal oxide **particles** and metal nitride **particles,** as well as to transition metal atoms, such as iron, cobalt, palladium and platinum.

Detailed Description Text - DETX (58):
   Even though the particles form loose agglomerates, the nanometer scale of
the primary particles is clearly observable in transmission electron
micrographs of the particles. The particles generally have a surface area
corresponding to particles on a nanometer scale as observed in the micrographs.
Furthermore, the particles can manifest unique properties due to their small
size and large surface area per weight of material. For example, vanadium
oxide **nanoparticles** can exhibit surprisingly high energy densities in lithium
batteries, as described in U.S. Pat. No. 5,952,125 to Bi et al., entitled
"Batteries With Electroactive **Nanoparticles,**" incorporated herein by reference.

Detailed Description Text - DETX (61):
   In addition, the **nanoparticles** preferably have a very high purity level.
The **nanoparticles** produced by laser pyrolysis are expected to have a purity
greater than the reactants because the laser pyrolysis reaction and, when
applicable, the crystal formation process tends to exclude contaminants from
the particle. Furthermore, crystalline **nanoparticles** produced by laser
pyrolysis have a high degree of crystallinity. Similarly, the crystalline
nanoparticles produced by heat processing have a high degree of crystallinity.
Impurities on the surface of the particles may be removed by heating the
particles to achieve not only high crystalline purity but high purity overall.

Detailed Description Text - DETX (63):
   A basic feature of successful application of laser pyrolysis for the
production of desirable inorganic **nanoparticles** is the generation of a reactant
stream containing a metal/metalloid precursor compound, a radiation absorber
and, generally, a secondary reactant. The secondary reactant can be a source
of atoms, such as oxygen, required for the desired product or an oxidizing or
reducing agent to drive a desired product formation. A secondary reactant is
not needed if the precursor decomposes to the desired product under intense
light radiation. Similarly, a separate radiation absorber is not needed if the
metal/metalloid precursor and/or the secondary reactant absorb the appropriate
light radiation. The reactant stream is pyrolyzed by an intense light beam,
generally a laser beam. As the reactant stream leaves the laser beam, the
particles are rapidly quenched.

Detailed Description Text - DETX (65):
   Nanoparticles produced by laser pyrolysis can be subjected to additional
processing to alter the nature of the particles, such as the composition and/or
the crystallinity. For example, the **nanoparticles** can be subjected to heat
processing in a gas atmosphere prior to use. Under suitably mild conditions,
heat processing is effective to modify the characteristics of the particles

without destroying the nanoscale size or the narrow particle size distribution of the initial particles. For example, heat processing of submicron vanadium oxide particles is described in U.S. Pat. No. 5,989,514 to Bi et al., entitled "Processing Of Vanadium Oxide Particles With Heat," incorporated herein by reference.

Detailed Description Text - DETX (67):
In particular, the production of vanadium oxide **nanoparticles** is described in U.S. Pat. No. 6,106,798 to Bi et al., entitled "Vanadium Oxide **Nanoparticles,**" incorporated herein by reference. Similarly, silver vanadium oxide **nanoparticles** have been produced, as described in copending and commonly assigned U.S. patent applications Ser. No. 09/246,076 to Horne et al., now U.S. Pat. No. 6,225,007 and Ser. No. 09/311,506, now U.S. Pat. No. 6,394,494 to Reitz et al., both entitled "Metal Vanadium Oxide Particles," both of which are incorporated herein by reference.

Detailed Description Text - DETX (69):
Furthermore, lithium manganese oxide **nanoparticles** have been produced by laser pyrolysis along with or without subsequent heat processing, as described in copending and commonly assigned U.S. patent applications Ser. No. 09/188,768, now U.S. Pat. No. 6,607,706 to Kumar et al., entitled "Composite Metal Oxide Particles," and 09/334,203, now U.S. Pat. No. 6,482,374 to Kumar et al., entitled "Reaction Methods for Producing Ternary Particles," and U.S. Pat. No. 6,136,287 to Horne et al., entitled "Lithium Manganese Oxides and Batteries," all three of which are incorporated herein by reference.

Detailed Description Text - DETX (70):
The production of silicon oxide **nanoparticles** is described in copending and commonly assigned U.S. patent application Ser. No. 09/085,514, now U.S. Pat. No. 6,726,990 to Kumar et al., entitled "Silicon Oxide Particles," incorporated herein by reference. This patent application describes the production of amorphous SiO.sub.2. The synthesis by laser pyrolysis of silicon carbide and silicon nitride is described in copending and commonly assigned U.S. patent application Ser. No. 09/433,202 to Reitz et al. filed on Nov. 5, 1999, entitled "Particle Dispersions," incorporated herein by reference.

Detailed Description Text - DETX (71):
The production of titanium oxide **nanoparticles** is described in copending and commonly assigned, U.S. patent application Ser. No. 09/123,255, now U.S. Pat. No. 6,387,531 to Bi et al., entitled "Metal (Silicon) Oxide/Carbon Composites," incorporated herein by reference. In particular, this application describes the production of anatase and rutile TiO.sub.2. The production of

aluminum oxide **nanoparticles** is described in copending and commonly assigned, U.S. patent application Ser. No. 09/136,483 to Kumar at al., entitled "Aluminum Oxide Particles," incorporated herein by reference. In particular, this application disclosed the production of $.gamma.-Al.sub.2 O.sub.3$. Suitable liquid, aluminum precursors with sufficient vapor pressure of gaseous delivery include, for example, aluminum s-butoxide (Al(OC.sub.4 H.sub.9).sub.3). Also, a number of suitable solid, aluminum precursor compounds are available including, for example, aluminum chloride (AlCl.sub.3), aluminum ethoxide (Al(OC.sub.2 H.sub.5).sub.3), and aluminum isopropoxide (Al[OCH(CH.sub.3).sub.2].sub.3).

Detailed Description Text - DETX (72):
   In addition, tin oxide **nanoparticles** have been produced by laser pyrolysis, as described in copending and commonly assigned U.S. patent application Ser. No. 09/042,227, now U.S. Pat. No. 6,200,674 to Kumar et al., entitled "Tin Oxide Particles," incorporated herein by reference. The production of zinc oxide **nanoparticles** is described in copending and commonly assigned U.S. patent application Ser. No. 09/266,202 to Reitz, entitled "Zinc Oxide Particles," incorporated herein by reference. In particular, the production of ZnO **nanoparticles** is described.

Detailed Description Text - DETX (73):
   The production of iron and iron carbide is described in a publication by Bi et al., entitled "Nanocrystalline .alpha.-Fe, Fe.sub.3 C, and Fe.sub.7 C.sub.3 produced by CO.sub.2 laser pyrolysis," J. Mater. Res. Vol. 8, No. 7 1666-1674 (Jul. 1993), incorporated herein by reference. The production of iron oxide **nanoparticles** is described in U.S. Pat. No. 6,080,337 to Kambe et al., entitled "Iron Oxide Particles," incorporated herein by reference. The production of **nanoparticles** of silver metal is described in copending and commonly assigned U.S. patent application Ser. No. 09/311,506, now U.S. Pat. No. 6,394,494 to Reitz et al., entitled "Metal Vanadium Oxide Particles," incorporated herein by reference.

Detailed Description Text - DETX (74):
   The production of iron sulfide (Fe.sub.1-x S) **nanoparticles** by laser pyrolysis is described in Bi et al., Material Research Society Symposium Proceedings, vol 286, p. 161-166 (1993), incorporated herein by reference. Precursors for laser pyrolysis production of iron sulfide were iron pentacarbonyl (Fe(CO).sub.5) and hydrogen sulfide (H.sub.2 S).

Detailed Description Text - DETX (76):
   The production of ternary **nanoparticles** of aluminum silicate and aluminum

titanate can be performed by laser pyrolysis following procedures similar to the production of silver vanadium oxide **nanoparticles** described in copending and commonly assigned U.S. patent application Ser. No. 09/311,506, now U.S. Pat. No. 6,394,494 to Reitz et al., entitled "Metal Vanadium Oxide Particles," incorporated herein by reference. Suitable precursors for the production of aluminum silicate include, for vapor delivery, a mixture of aluminum chloride (AlCl.sub.3) and silicon tetrachloride (SiCl.sub.4) and, for aerosol delivery, a mixture of tetra(N-butoxy) silane and aluminum isopropoxide (Al(OCH(CH.sub.3).sub.2).sub.3). Similarly, suitable precursors for the production of aluminum titanate include, for aerosol delivery, a mixture of aluminum nitrate (Al(NO.sub.3).sub.3) and titanium dioxide (TiO.sub.2) powder dissolved in sulfuric acid or a mixture of aluminum isopropoxide and titanium isopropoxide (Ti(OCH(CH.sub.3).sub.2).sub.4).

Detailed Description Text - DETX (78):
   To form the composites, generally the inorganic particles are dispersed in a liquid and combined with polymer/monomers constituents and the linker. The formation of the particle dispersion generally is a distinct step of the process. Preferably, a collection of **nanoparticles** is well dispersed for uniform introduction into a polymer composite. A liquid phase particle dispersion can provide a source of small secondary particles that can be used in the formation of desirable composite structures.

Detailed Description Text - DETX (81):
   The composition of the dispersion depends on the composition of the dispersant and the **nanoparticles**. Suitable dispersants include, for example, water, organic solvents, such as alcohols and hydrocarbons, and combinations thereof. The selection of preferred solvents generally depends on the properties of the **nanoparticles**. Thus, the dispersant and the **nanoparticles** should be selected to be compatible for the formation of well dispersed particles. For example, gamma alumina particles generally are dispersed well at acidic pH values of about 3-4, silica particles generally are dispersed well at basic pH values from 9-11, and titanium oxide particles generally disperse well at a pH near 7, although the preferred pH depends on the crystal structure and the surface structure. Generally, **nanoparticles** with little surface charge can be dispersed preferentially in less polar solvents. Thus, hydrophobic particles can be dispersed in nonaqueous solvents or aqueous solutions with less polar cosolvents, and hydrophilic particles can be dispersed in aqueous solvent.

Detailed Description Text - DETX (87):
   The secondary particle size refers to the size of the resulting particle

agglomerates following dispersion of the powders in the liquid. Smaller secondary particles sizes are obtained if there is more disruption of the agglomerating forces between the primary particles. Secondary particles sizes equal to the primary particle sizes can be accomplished with at least some **nanoparticles** if the interparticle forces can be sufficiently disrupted. The use of surfactants and high shear stress can assist with obtaining smaller secondary particle sizes.

Detailed Description Text - DETX (101):
The inorganic particles can be associated with only one of the polymer compositions within the block such that the inorganic particles are segregated together with that polymer composition within the segregation block copolymer. For example, an AB di-block copolymer can include inorganic particles only within block A. Segregation of the inorganic particles can have functional advantages with respect to taking advantage of the properties of the inorganic particles. Similarly, tethered inorganic particles may separate relative to the polymer by analogy to different blocks of a block copolymer if the inorganic particles and the corresponding polymers have different solvation properties. In addition, the **nanoparticles** themselves can segregate relative to the polymer to form a self-organized structure.

Detailed Description Text - DETX (103):
Regardless of the self-organization mechanism, some self-organized composites involve **nanoparticles** aligned with periodicity in a superstructure or super crystal structure. The particles may or may not be crystalline themselves yet they will exhibit properties due to the ordered structure of the particles. Photonic crystals make use of these crystal superstructures, as described further below.

Detailed Description Text - DETX (106):
In some embodiments involving self-assembly with **nanoparticles,** a portion of the substrate surface is provided with pores, which can be holes, depressions, cavities or the like. The pores can be in an ordered array or a random arrangement. The size of the pores should be larger than the size of the **nanoparticles**. Generally, the pores have a diameter less than a micron, although the preferred size of the pores and density of the pores may depend on the particular desired properties of the resulting device.

Detailed Description Text - DETX (117):
The polymer/inorganic particle composite materials are suitable for the efficient formation of devices incorporating a very wide range of materials. The composites can incorporate preferably one or more of the various very

uniform **nanoparticles** that have been described above. Selective incorporation
of particular composites into a particular device can establish desired
function for a device due to the choice of composite.

Detailed Description Text - DETX (119):
    Examples of structures placed along a substrate incorporating
polymer/inorganic particle composites are shown in FIG. 10. Referring to FIG.
10, a substrate 200 includes structures or islands 202, 204, 206, 208, 210, 212
with composite material. Integrated self assembled structures are described
further in copending and commonly assigned U.S. patent application Ser. No.
09/558,266 to Kambe et al., entitled "Self Assembled Structures," incorporated
herein by reference. Each of the composites in structures 202, 204, 206, 208,
210, 212 can include the same composition or a different polymer composition
and/or inorganic particles as the other structures. Preferred nanoscale
particles have a narrow particle size distribution of primary particles, such
as the **nanoparticles** described above formed by laser pyrolysis. Similarly,
preferred nanoscale particles have a small average secondary particle size,
generally resulting from the use of a preferred particle dispersion.

Detailed Description Text - DETX (120):
    Suitable devices incorporating **nanoparticles** or other self-assembled
compositions include, for example, energy sources, such as batteries; photonic
crystals; active electrical or electro-optical elements, such as field emission
devices; and passive elements, such as electrical interconnects, barrier layers
and insulating layers. Electrodes can be formed with self-assembled
electroactive particles along with electrically conductive particles.
Similarly, the electrodes can be formed with electrically conducting polymers
and suitable inorganic particles to form the composite.

Detailed Description Text - DETX (121):
    Photonic crystals are ordered arrays of composite composition having a unit
cell size of the photonic crystal ranging from about one quarter to about one
optical wavelength. The index-of-refraction of the material depends on the
wavelength of light. For example, visible light in air has a wavelength of
about 380 nm to about 780 nm. Generally, photonic crystals of interest have
size from about 100 nm to about 1000 nm. The particles form a crystal
superstructure with alternating regions of index-of-refraction. The photonic
crystals can be formed from an ordered array of **nanoparticles** of, for example,
metals, silica, silicon nitride, silicon oxynitride, titania or zinc oxide.
Due to the size of the ordered arrays, the photonic crystals can have a
photonic band gap that prevents propagation of light in any direction. Thus,
photonic crystals can be used for control of spontaneous emission and for very

sharp bending of light. Self-assembly, as described above, can be used to form the ordered arrays.

Detailed Description Text - DETX (122):

Electrical interconnects can be constructed from electrically conductive **particles,** for example, metal **nanoparticles,** such as silver and **gold nanoparticles**. Similarly, optical interconnects provide for transmission of light between devices. Integrated optical interconnects can be formed from materials with suitable indices of refraction. For transmitting visible light, silica, alumina and zinc oxide, for example, can be used. Barrier layers can be formed, for example, from silicon oxide **particles** under higher index-of-refraction materials. Insulating layers can be formed, for example, from silicon dioxide **nanoparticles**. Field emission devices for displays can incorporate phosphor **particles,** such as zinc oxide or doped zinc oxide.

Detailed Description Text - DETX (140):

An x-ray diffractogram of product **nanoparticles** produced under the conditions in Table 1 are shown in FIG. 17. Sample TiO_2 -1 had an x-ray diffractogram corresponding to rutile TiO_2. Sample TiO_2 -2 had an x-ray diffractogram similar to sample TiO_2 -1. Sample TiO_2 -3 had an x-ray diffractogram corresponding to anatase TiO_2. The broadness of the peaks in FIG. 17 indicates that sample 1 is less crystalline than the other two samples. Some peaks in the spectra of sample TiO_2 -1 seem to originate from amorphous phases.

Detailed Description Text - DETX (143):

This example provides a description of the formation of well dispersed dilute solutions of titanium oxide **nanoparticles** produced by laser pyrolysis, as described in Example 1.

Detailed Description Text - DETX (148):

Since the particle size analyzer had a detection cut-off at 30 nm, an internal standard was used to estimate the number of particles with diameters less than 30 nm. A well characterized commercial TiO_2 powder (R706 average particle size 0.36 microns with a coating of Al_2O_3, DuPont, Wilmington, Del.) was mixed with the **nanoparticles** in a 1 to 1 by weight ratio. The resulting suspension was analyzed with the Horiba particle size analyzer. Less than about 10 percent of the **nanoparticles** were observed. Thus, most of the **nanoparticles** had a diameter less than about 30 nm and are undetected by the particle size analyzer. Nevertheless, the trends measured with the particle size analyzer were indicative of the level of agglomeration. In particular, good dispersions were formed with solvents that performed well in

suspending the particles.